UNIVERSITY OF SWAZILAND
SECOND SEMESTER EXAMINATION 2009/2010

TITLE OF PAPER	:	Instrumental Methods For Environmental Analysis - 2
COURSE NUMBEER	:	EHS 574
TIME ALLOWED	:	Two(2) Hours
INSTRUCTIONS	:	Answer any four (4) questions. Each Question carries 25 marks.

A periodic table and other useful data have been provided with this paper.

You are not supposed to open this paper until permission to do so has been granted by the Chief Invigilator.

Question 1(25 marks)

(a)   What is an internal standard?   [1]

(b)   Describe the graphical method of the standard addition method in AAS/FES during quantitative elemental analysis. Give one unique advantage of this method.   [7]

(c)   Why is an internal standard most appropriate for quantitative analysis when unavoidable losses of analytes are expected during sample preparation?

[2]

(c)   Aliquots of the standard solution of an element X were mixed with an unknown sample containing X for AAS analysis. The standard solution contained 1.00mg of X per liter. The following absorbance readings were obtained:

Vol. of Unknown (mL)	Vol. of standard (mL)	Total Volume (mL)	Absorbance
10.00	0	100.00	0.163
10.00	1.00	100.00	0.240
10.00	2.00	100.00	0.319
10.00	3.00	100.00	0.402
10.00	4.00	100.00	0.478

(i)   Calculate the concentration (in mg/L) of added standard to each solution.   [5]

(ii)   Using graphical method, determine the concentration of X in the unknown.   [10]

Question 2(25 marks)

a)   What is solvent extraction?   [1]

b)   Define K (the distribution coefficient) and D (the distribution ratio). State any difference/s between them.   [4]

c)   Briefly describe the procedure for the extraction of a solute contained in a 50.0mL aqueous phase using 100.0mL carbon tetrachloride   [7]

d)   Using an appropriate expression. identify the factors that influence the distribution ratio (D) of an acid that is monomeric in both aqueous and organic phases and whose anion does not penetrate the organic layer.   [4]

e) i) Ninety percent of a certain solute is extracted when equal volumes of aqueous and organic phases are used. What will be the percent extracted if the volume of the organic phase is doubled? [6]

ii) If, instead of doubling the volume of the organic phase, the extraction is carried out twice using the same volume of the organic phase, which of the two procedure would you prefer and why? [3]

Question 3(25 marks)

a) Define the following chromatographic terms:
   i) Retention, time t_R
   ii) Retention volume, V_R [2]

b) Using an illustrative choromatogram, discuss how chromatographic methods can be employed for both quantitative and qualitative analysis of a sample. [7]

c) Draw and label the schematic diagram of a 'Gas Chromatograph' (GC) [4]

d) For the GC, discuss :
   i) The main features of a packed column. [4]
   ii) The function and the ideal properties of the solid support for the column. [4]
   iii) The function and the ideal properties of the liquid phase for the column. [4]

Question 4(25 marks)

a) In gas chromatography (GC) what is column efficiency? How is its value affected by N, the number of theoretical plates, and H, the plate height? What other factors affect it? [5]

b) What is temperature programming in GC? Use a graphical illustration to show how it affects the resolution, R the retention time, t_r and the number of solutes eluted during a GC analysis. What are its advantages over the isothermal procedure? [11]

c) Give five general applications of 'Gas Chromatographic analysis'. Give four examples of an industries and laboratories in Swaziland where this method is being used on routine basis. [9]

Question 5(25 marks)

a)   Distinguish between 'Thin Layer Chromatography' (TLC) and 'Paper
        Chromatography' from the following points of view:
     i) the nature of the phase.
     ii) the nature of the stationary phase
     iii) resolution and sensitivity.   [6]

b) Define R_f value, with regards to qualitative analysis in planar chromatography.
                                                                   [2]

c)   For the analysis of a polar substance using the TLC method, give a brief
        procedure for the:
     i) TLC plate preparation   [8]
     ii) Identification of the separated components on the TLC plate.   [9]

PERIODIC TABLE OF ELEMENTS

GROUPS

PERIODS	1 IA	2 IIA	3 IIIB	4 IVB	5 VB	6 VIB	7 VIIB	8 VIIIB	9 VIIIB	10 VIIIB	11 IB	12 IIB	13 IIIA	14 IVA	15 VA	16 VIA	17 VIIA
1	1.008 H 1																
2	6.941 Li 3	9.012 Be 4											10.811 B 5	12.011 C 6	14.007 N 7	15.999 O 8	18.998 F 9
3	22.990 Na 11	24.305 Mg 12											26.982 Al 13	28.086 Si 14	30.974 P 15	32.06 S 16	35.45 Cl 17
4	39.098 K 19	40.078 Ca 20	44.956 Sc 21	47.88 Ti 22	50.942 V 23	51.996 Cr 24	54.938 Mn 25	55.847 Fe 26	58.933 Co 27	58.69 Ni 28	63.546 Cu 29	65.39 Zn 30	69.723 Ga 31	72.61 Ge 32	74.922 As 33	78.96 Se 34	79.90 Br 35
5	85.468 Rb 37	87.62 Sr 38	88.906 Y 39	91.224 Zr 40	92.906 Nb 41	95.94 Mo 42	98.907 Tc 43	101.07 Ru 44	102.91 Rh 45	106.42 Pd 46	107.87 Ag 47	112.41 Cd 48	114.82 In 49	118.71 Sn 50	121.75 Sb 51	127.60 Te 52	126.9 I 53
6	132.91 Cs 55	137.33 Ba 56	*138.91 La 57	178.49 Hf 72	180.95 Ta 73	183.85 W 74	186.21 Re 75	190.2 Os 76	192.22 Ir 77	195.08 Pt 78	196.97 Au 79	200.59 Hg 80	204.38 Tl 81	207.2 Pb 82	208.98 Bi 83	(209) Po 84	(210) At 85
7	223 Fr 87	226.03 Ra 88	**(227) Ac 89	(261) Rf 104	(262) Ha 105	(263) Unh 106	(262) Uns 107	(265) Uno 108	(266) Une 109	(267) Uun 110							

TRANSITION ELEMENTS

Key:
Atomic mass → 10.811
Symbol → B
Atomic No. → 5

(example: 26.982 Al 13)

*Lanthanide Series

140.12 Ce 58	140.91 Pr 59	144.24 Nd 60	(145) Pm 61	150.36 Sm 62	151.96 Eu 63	157.25 Gd 64	158.93 Tb 65	162.50 Dy 66	164.93 Ho 67	167.26 Er 68	168.93 Tm 69	173.04 Yb 70	174.9 Lu 71

**Actinide Series

232.04 Th 90	231.04 Pa 91	238.03 U 92	237.05 Np 93	(244) Pu 94	(243) Am 95	(247) Cm 96	(247) Bk 97	(251) Cf 98	(252) Es 99	(257) Fm 100	(258) Md 101	(259) No 102	(260) Lr 103

( ) indicates the mass number of the isotope with the longest half-life.

Quantity	Symbol	Value
Speed of light†	c	$2.997\,924\,58 \times 10^8$ m s^{-1}
Elementary charge	e	$1.602\,177 \times 10^{-19}$ C
Faraday constant	$F = eN_A$	9.6485×10^4 C mol^{-1}
Boltzmann constant	k	$1.380\,66 \times 10^{-23}$ J K^{-1}
Gas constant	$R = kN_A$	$8.314\,51$ J K^{-1} mol^{-1}
		$8.205\,78 \times 10^{-2}$ dm^3 atm K^{-1} mol^{-1}
		62.364 L Torr K^{-1} mol^{-1}
Planck constant	h	$6.626\,08 \times 10^{-34}$ J s
	$\hbar = h/2\pi$	$1.054\,57 \times 10^{-34}$ J s
Avogadro constant	N_A	$6.022\,14 \times 10^{23}$ mol^{-1}
Atomic mass unit	u	$1.660\,54 \times 10^{-27}$ kg
Mass of electron	m_e	$9.109\,39 \times 10^{-31}$ kg
proton	m_p	$1.672\,62 \times 10^{-27}$ kg
neutron	m_n	$1.674\,93 \times 10^{-27}$ kg
Vacuum permeability†	μ_0	$4\pi \times 10^{-7}$ J s^2 C^{-2} m^{-1}
		$4\pi \times 10^{-7}$ T^2 J^{-1} m^3
Vacuum permittivity	$\varepsilon_0 = 1/c^2\mu_0$	$8.854\,19 \times 10^{-12}$ J^{-1} C^2 m^{-1}
	$4\pi\varepsilon_0$	$1.112\,65 \times 10^{-10}$ J^{-1} C^2 m^{-1}
Bohr magneton	$\mu_B = e\hbar/2m_e$	$9.274\,02 \times 10^{-24}$ J T^{-1}
Nuclear magneton	$\mu_N = e\hbar/2m_p$	$5.050\,79 \times 10^{-27}$ J T^{-1}
Electron g value	g_e	$2.002\,32$
Bohr radius	$a_0 = 4\pi\varepsilon_0\hbar^2/m_e e^2$	$5.291\,77 \times 10^{-11}$ m
Rydberg constant	$R_\infty = m_e e^4/8h^3c\varepsilon_0^2$	$1.097\,37 \times 10^5$ cm^{-1}
Fine structure constant	$\alpha = \mu_0 e^2 c/2h$	$7.297\,35 \times 10^{-3}$
Gravitational constant	G	$6.672\,59 \times 10^{-11}$ N m^2 kg^{-2}
Standard acceleration of free fall†	g	$9.806\,65$ m s^{-2}

General data and fundamental constants

† Exact (defined) values

f	p	n	μ	m	c	d	k	M	G	Prefixes
femto	pico	nano	micro	milli	centi	deci	kilo	mega	giga	
10^{-15}	10^{-12}	10^{-9}	10^{-6}	10^{-3}	10^{-2}	10^{-1}	10^3	10^6	10^9	